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Final Report

on NASA Grant NSG-8029

Determination of Particle Size Using Measurement of Scatter

To

National Aeronautics and Space Administration

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Summary

Measurements of scatter from glass beads placed on a mirror were taken using a quartz lamp and laser. Data were obtained in the plane containing the normal to the surface and the incident beam. For the quartz lamp the best correlation can be obtained for data measured at a Zenith of 55^{0} and 180^{0} from the incident beam. The best correlation when the laser is used is obtained at about the same position. However, the correlation variables are angular spacing of the minimums of the data versus the size. Whereas, with the quartz lamp the correlation variables are the amount of scattered energy versus size.

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Introduction

One phenomenon which may occur during space flight is contamination of the spacecraft with particles of unknown materials with unknown sizes. A contamination of the spacecraft may cause a component failure that affects the spacecraft mission. This investigation is concerned with determining by experimental means if glass spherical particle contamination can be identified and sized by use of light scatter from the particles.

Figure 1 shows the schematic arrangement for the experiment. The glass bead is placed on an aluminum front surface mirror and illuminated with light. Efforts were made using a laser, monochromator, and quartz lamp with no monochromator. Measurements of scattered light was accomplished with a 1P28A photomultiplier tube.

The detector Zenith θ , can be varied from 0-85° and the detector motion about the normal to the mirror (it's Azimuth θ) can be varied from 0-360°. A detector of 0° means the detector is in the plane of the normal and the incident beam and 180° from the source.

The reflected light is composed of a very strong beam at the same Zenith as the incident beam and an Azimuth of 0°, and scattered light in all directions regardless of

whether a beam is or is not on the mirror. The goal of this investigation is to determine if measurements of scatter taken for beads whose size range from 50-136 microns can be correlated to the size of the bead.

There are several reasons why it is difficult to obtain accurate data for the stated problem. These are:

1. For small beads mirror scatter becomes important so that mirror scatter needs to be measured accurately. However, the mirror scatter is difficult to measure accurately because fine particles from the room settle on the mirror and the mirror reflectance has been found to vary from point to point on the mirror. Also the mirror cleaning procedure may cause some deviation of it's reflectance.
2. The beads may not be true spheres.
3. The index of refraction may vary from bead to bead and possibly cause a variation in scatter that is not related to size. This possible variation of the index of refraction can be seen by viewing the beads through the microscope. Some beads are glassy looking, some are black and some(most) are white looking when the illumination is normal to the mirror.

4. The surface of the beads varied from bead to bead possibly causing a variation in scatter.
5. The light energy in the source may vary across the cross section of the beam, making it difficult to establish a procedure for placing a bead under the source.
6. Accurate measurements of the diameter of the beads are difficult to make.

Given the goal of the investigation and the difficulties involved, it was decided to choose an experimental arrangement that appeared promising then work on eliminating the difficulties. The optical arrangement that was tried used the laser, monochromator with quartz lamp and quartz lamp.

One of the requirements of the incident light is a small cross sectional area of the beam at the point of impingement on the glass bead. The laser is ideally suited for this because the beam due to its parallel nature can be focused to any cross sectional area by suitable lens or mirrors. Because of this and the more than adequate amount of energy in the beam the laser was tried first. Figure 3 shows a photograph of the scattered energy and Figure 4 shows some data taken with the laser. The film was located approximately 15cm from the bead in the plane containing the normal to the surface and the incident ray, and 40° from the normal.

When a large solid angle is used as was the case with the data of Figure 4 the results do not correlate. However when a small solid angle is used there is a correlation between spacing of lines shown in Figure 3 and the size of the beads.

An effort was made using the monochromator with the quartz lamp. This effort was unsuccessful because the energy level in the incident light was too low and the cross sectional area of the light ray was too large.

The last optical arrangement tried consisted of using the quartz lamp without the monochromator or any filter needed to get monochromatic light. After several initial runs were made it was apparant that there was a good possibility of at least establishing that size could be correlated with the light reflected from the beads. The obvious disadvantage of this arrangement is that any data taken is valid only for the photomultiplier tube and quartz lamp used.

Apparatus and Procedures

The bidirectional apparatus for measuring the scatter in this investigation was presented in NASA Grant NGR 19-005-009.

The detector output is proportional to the energy scattered from the mirror. The data presented here is the detector signal which is denoted by:

$$D(\psi,\zeta;\theta,\emptyset)=D_{meas}(\psi,\zeta;\theta,\emptyset)/\text{ Source}$$

where:

ψ=Source Zenith,

ζ=Source Azimuth,

θ=Detector Zenith,

$\emptyset$=Detector Azimuth.

D_{meas}=Actual Measurement from Detector

Source=Output for the source.

Essentially dividing the detector measurements of scatter by the source measurement corrects for source drift. The source was found to drift approximately 2 percent for the final measurements.

Some data is presented as

$$D_{bead}(\psi,\zeta;\theta,\emptyset)-D_{mirror}(\psi,\zeta;\theta,\emptyset)$$

which is the scatter with mirror and bead minus scatter for the mirror at the same angle. This was done primarily to show the importance of the mirror scatter in any correlation that may be obtained.

The microbeads used were Type A Class 5 beads obtained

from the Catophote Division of Ferro Coporation. As mentioned in the introduction the beads appeared to be of three types. In this investigation an attempt was made to use only the beads that were white looking, round, and relatively free of surface imperfections. These were sized by comparing a photograph of the beads and a photograph of a stage micrometer taken through the microscope. A typical photograph is shown in Figure 2. The beads were also sized by using a calibrated eyepiece micrometer. During the early period of the investigation the specimen bead was placed on the mirror by pouring many beads on the mirror then removing all beads except the one needed for the test. It was soon discovered that the procedure left many micron sized particles on the mirror which affected the reflectance of the mirror. For the final data the specimen bead was selected from a few beads placed on the mirror.

In order to remove any small particles a lens cleaning tissue was used to wipe the mirror before each test. The scatter at a Zenith of 0° was also measured before and after each test of a specimen bead. These two procedures were used to be certain the scatter remained the same before and after the test.

Placing the microbead under the incident light is perhaps the most important step in taking this data. For the first data taken, this was done by maneuvering the bead

with a horse hair until it was under the light. With this technique it was difficult to obtain repeatable data. Because of this, the specimen holder was modified to allow motion of the mirror perpendicular to the direction of the light beam. After this modification the bead was positioned by maneuvering it to maximize the signal at a detector position normal to the mirror. This improved the repeatability of the data.

Presentation and Discussion of Results
Quartz Lamp

The data of this investigation are given in Figures 4 through 13 and Tables 1 through 12. Data were taken for $\psi=30^\circ$, $\zeta=180^\circ$, $\phi=180^\circ$, $\phi=0^\circ$, and $\theta=0^\circ$-85°. Five sets of data were taken as follows:

1. $\phi=0^\circ$, $\theta=0^\circ$-20°
2. $\phi=180^\circ$, $\theta=0^\circ$-20°
3. $\phi=0^\circ$, $\theta=37.5^\circ$-20°
4. $\phi=180^\circ$, $\theta=37.5^\circ$-75°
5. $\phi=180^\circ$, $\theta=0^\circ$ and 20°

After preliminary data were taken it appeared that the data in set 2 were the most likely to correlate. Figure 6 shows data taken for this set where mostly the maximum and minimum were recorded. As a whole the detector signal seems to increase as the size of the bead increases. However, there is no apparent correlation among the maxima of the various curves. But, this data and preliminary data show the point $\theta=0^\circ$ to be special in that it is usually a point where the detector signal is a minimum.

Next a decision was made to take a large number of data points at $\theta=0^\circ$, however, it was discovered that only a small number of sizes were available. For the sizes that were available the results are shown in Figure 7. Similar type data are shown in Figure 8 for $\theta=20^\circ$ and $\phi=180^\circ$. Both curves show a trend of increasing scatter with particle size. Figure 8 and preliminary data indicates

that the detector signal changes fairly large with size for θ near the source incident angle. This is why a decision was made to take data sets 1, 3, and 4. Set 4 was taken first as is shown in Figure 9. The aim here was to see how much the detector signal, at about 40°, varied with size. This figure shows there is a considerable variation of signal with size but also a fast change in signal with angle is shown. This means that these points near 40° probably have a greater margin of error due to instrument inaccuracies in angle setting and solid angle resolution. Figure 5 shows that the mirror scatter at these angles is small so that the mirror error in any correlation obtained near 40° would be small. For instance for a 55.2 micron bead at ϕ=180°, D_{bead}=1.000 and D_{mirror} is approximately 0.17. This means this set of data does not require a very accurate measurement of the mirror scatter since an error of 10% in the mirror data causes an error of 0.017 or about 2% in the measurement for the bead.

The data of sets 1 and 3 are shown in Figures 10 and 11. Figure 11 for set 3 shows a good correlation between size and scatter. However it must be pointed out that the mirror causes considerable scatter near θ=40°. Also there is a sharp change in mirror scatter with θ near 40° which increases the margin of error caused by error in setting the angle during a test. It seems as if a good correlation exist for θ approximately equal 55° since at this

angle the mirror scatter is small compared to the bead scatter. In this case a 10% error in mirror scatter measurements results in a 1% error in bead scatter data for the smallest bead.

Figure 12 shows the data for set 3 at 52.5° with scatter plotted against size. For the five runs taken this shows a good correlation. As with the data at $\theta=20^{\circ}$, $\phi=180^{\circ}$ it is necessary to take data for many sizes to obtain a true correlation. This as before is because of the error involved in a single measurement.

Laser

As mentioned in the introduction the results for the laser when the detector subtends a large solid angle were not encouraging. However, when a solid angle small enough to discern the small lines in Figure 3 is used, there is a correlation between the angular spacing of the lines and the size of the particle as shown in Figure 13.

Conclusion

Figures 3, 12, and 13 are presented as preliminary correlations of size with scatter. It is anticipated that the procedures and apparatus will be improved for the second year of this investigation. Therefore the data of these figures will be taken again with a larger number of sizes.

Error Analysis

An error analysis of the measurement system when used to measure a Lambertian surface was presented in the final report to NASA Grant NGR 19-005-009. The error for such a surface was found to be approximately 1.15%. The additional causes of errors were given in the introduction. The error in the measurement of the diameter of the bead which is approximately 5% is the only one a reasonable estimate can be obtained for. As mentioned above an assumed error of 10% in the mirror reflections would cause an error of only 2% maximum for the recommended correlation curves. These errors give a combined error of 5.39%.

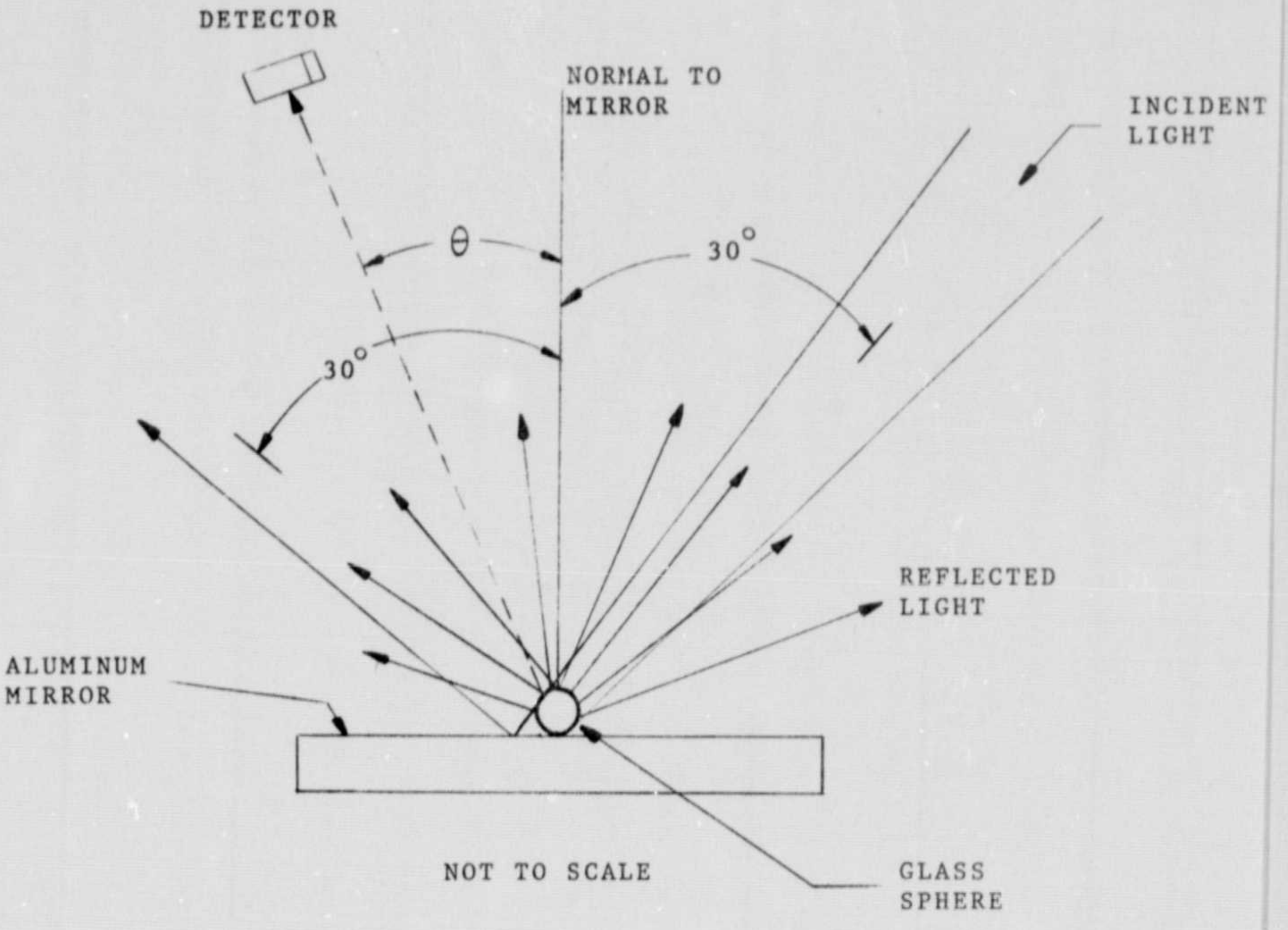

FIGURE 1 Schematic of specimen, light illumination, and detector arrangement.

Figure 2. Glass Bead and Stage Microscope at 300 Magnification; smallest stage division is 10 microns.

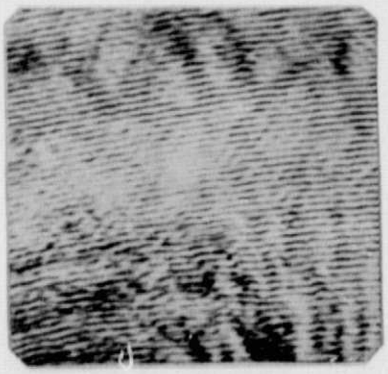

Figure 3. Photograph of Scattered Energy at $\theta=40^0$, $\emptyset=0^0$, and 15cm from specimen.

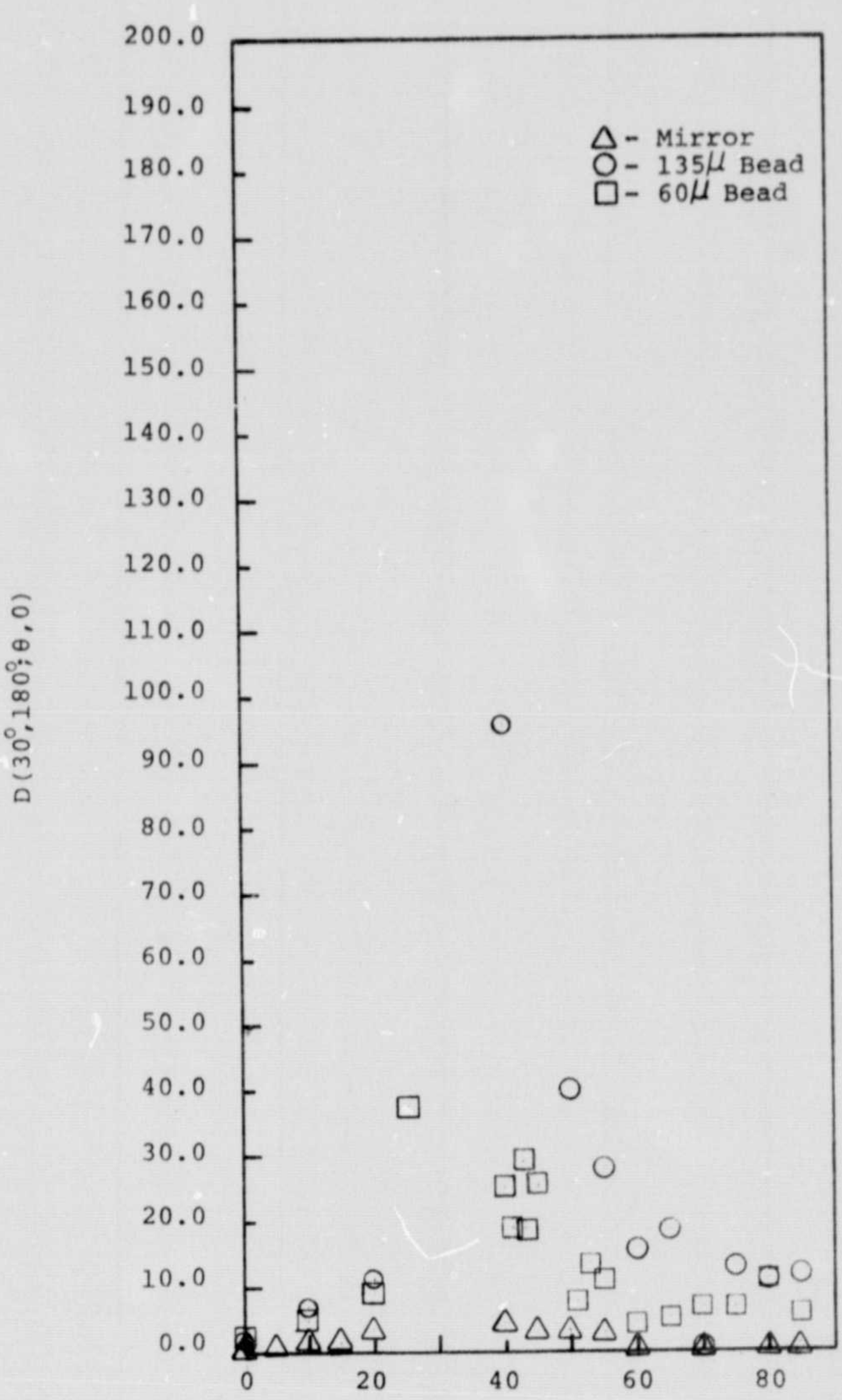

Figure 4 Reflectance at 0.6238μ , ϕ=0°

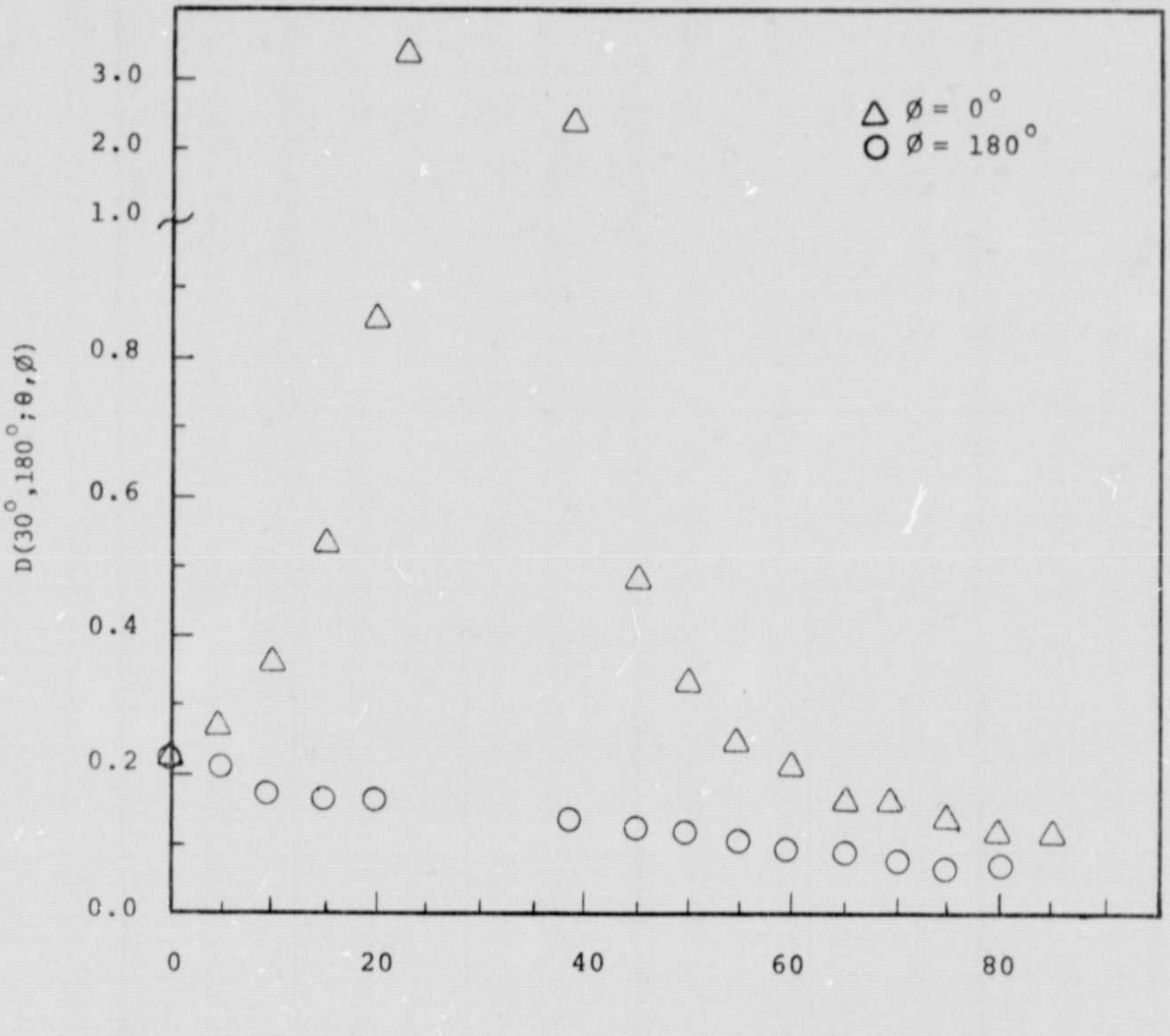

Figure 5 Reflectance of Mirror.

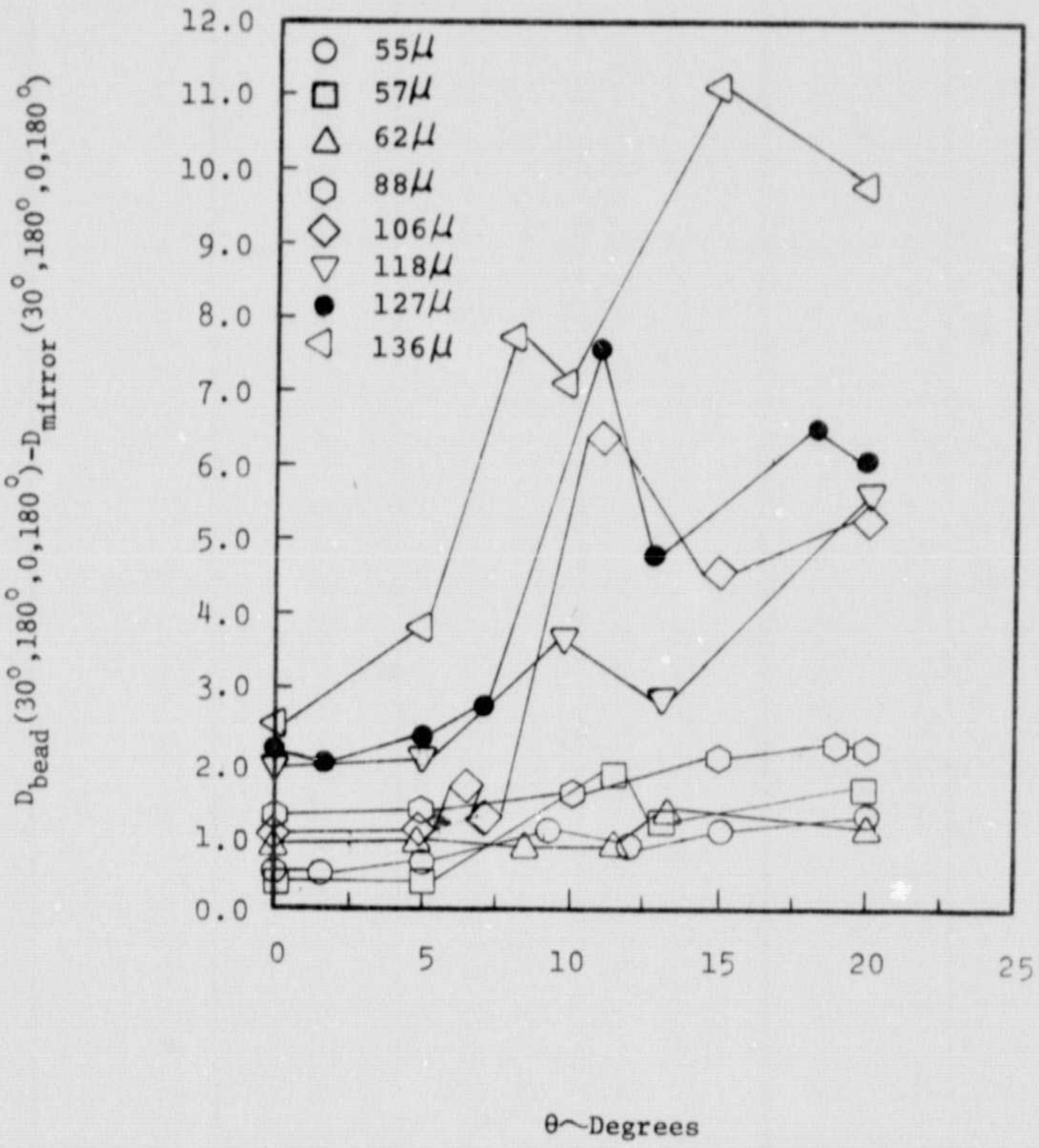

Figure 6 Measurement of Scatter for θ=0° to 20°, Ø=180°.

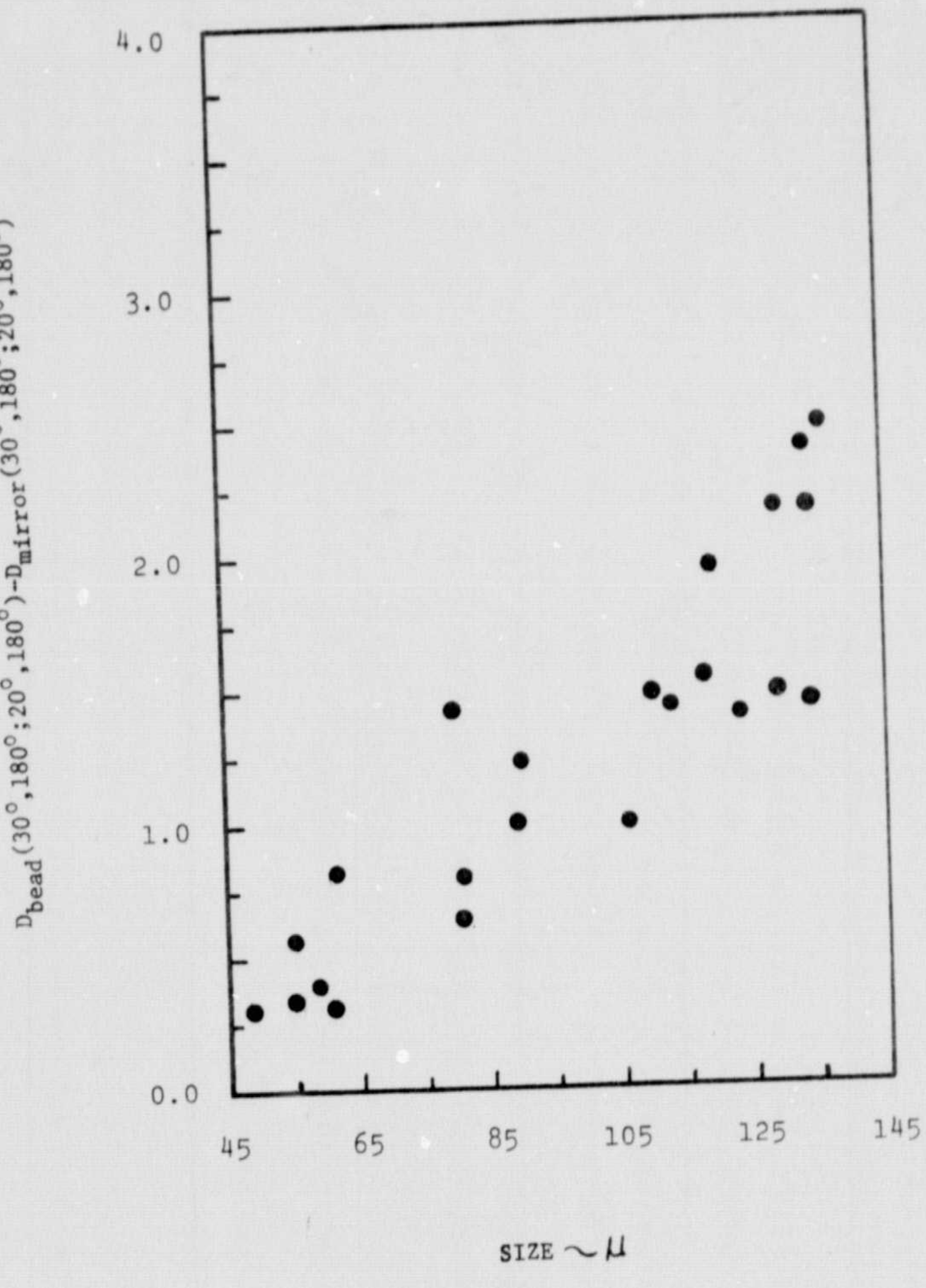

Figure 7 Measurements of Scatter for $\theta=0^\circ$.

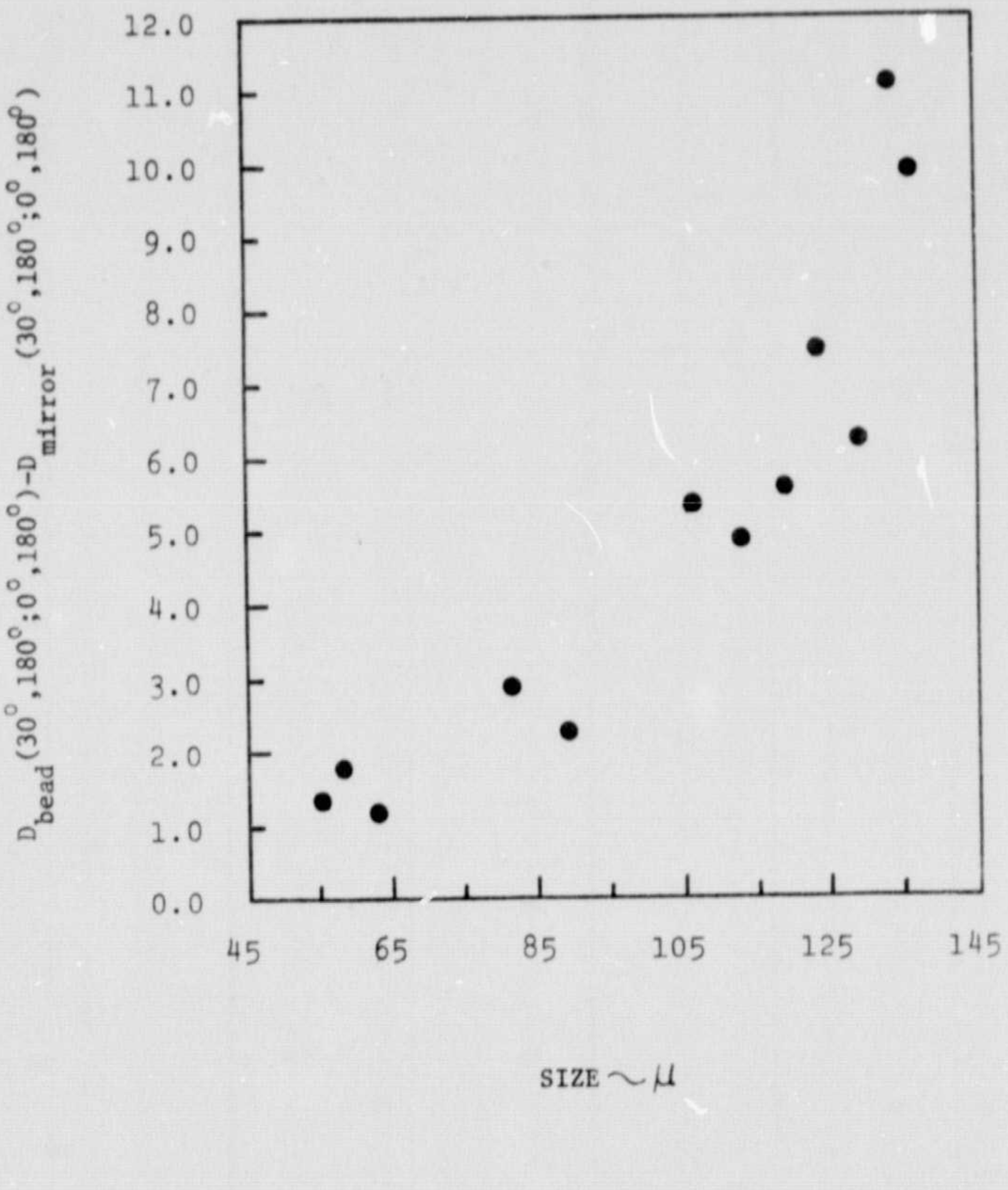

Figure 8 Measurements of Scatter for $\theta=20^\circ$, $\emptyset=180^\circ$.

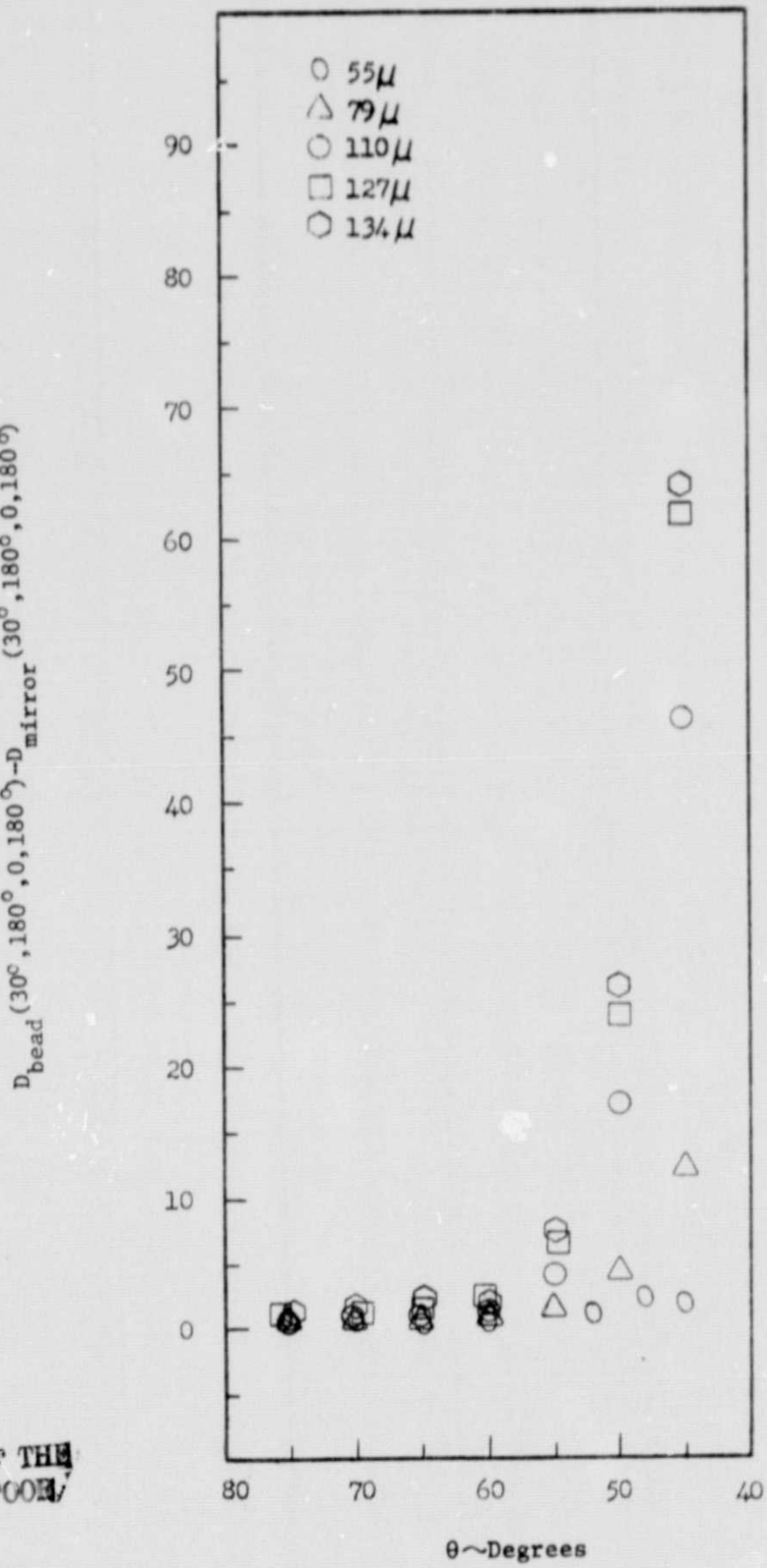

Figure 9 Measurements of Scatter for θ 40°, Ø=180°.

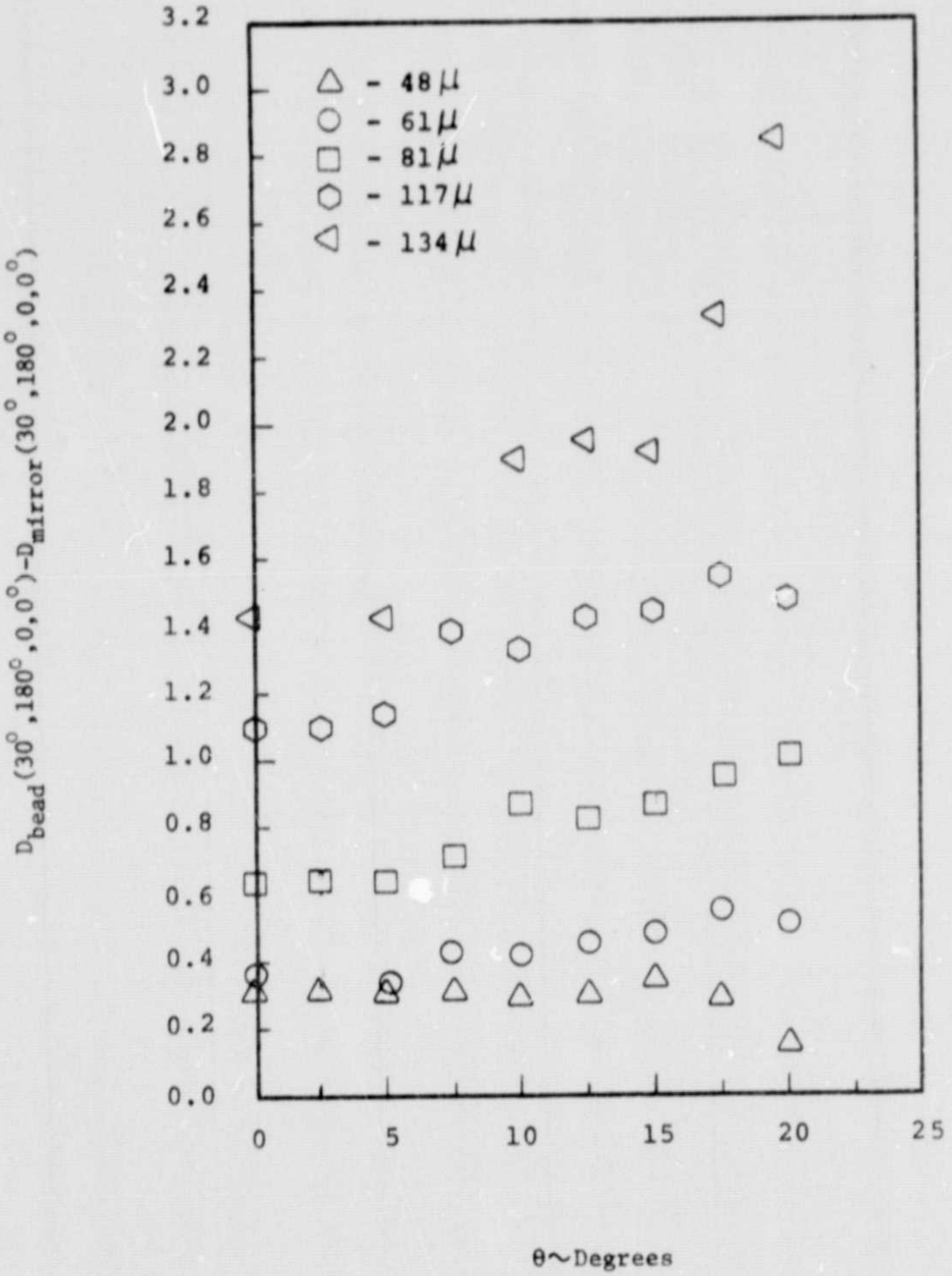

Figure 10 Measurements of Scatter for $\theta=0^\circ$ to 20° and $\phi=0^\circ$.

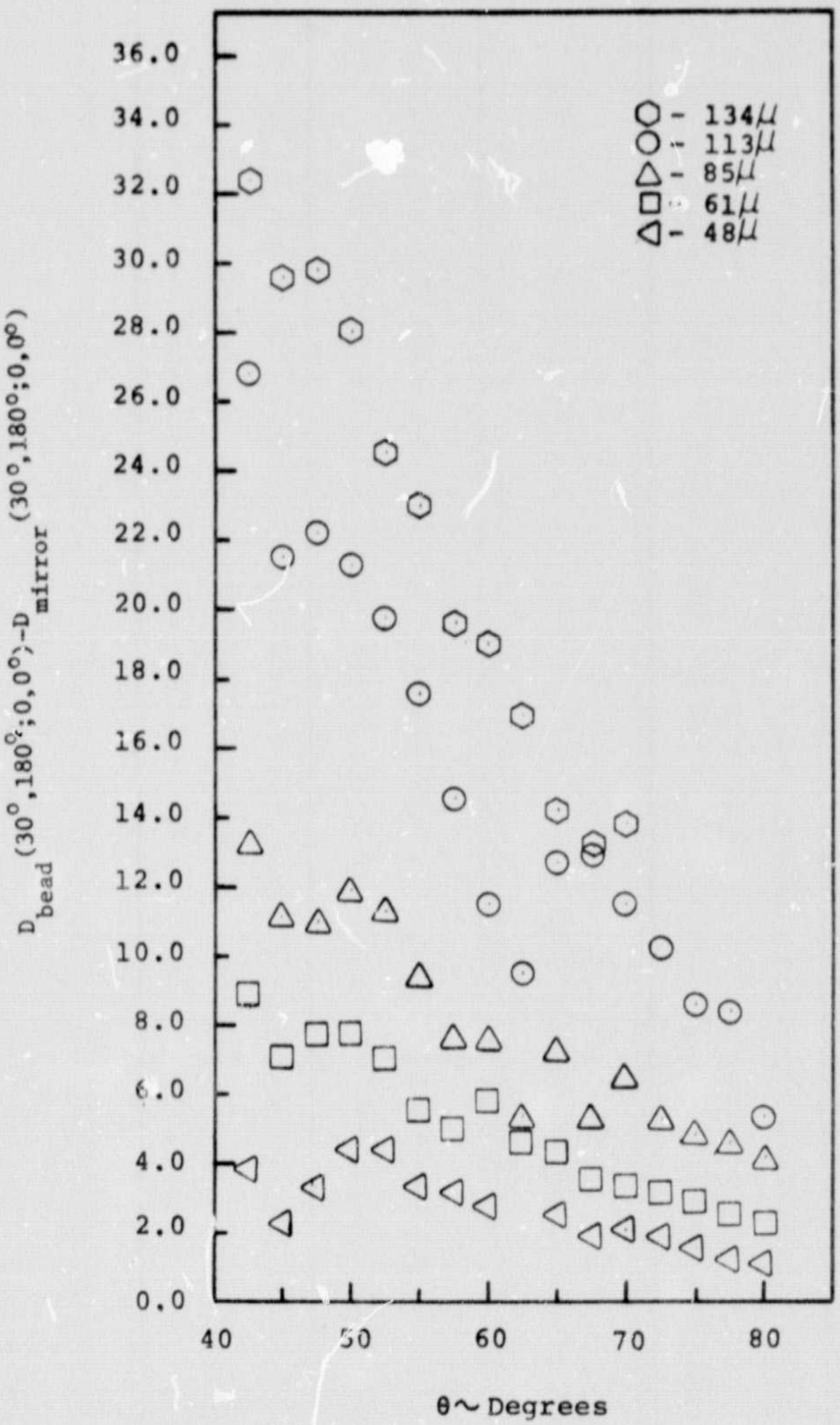

Figure 11 Measurements of Scatter for $\theta > 35°$ and $\varnothing=0°$.

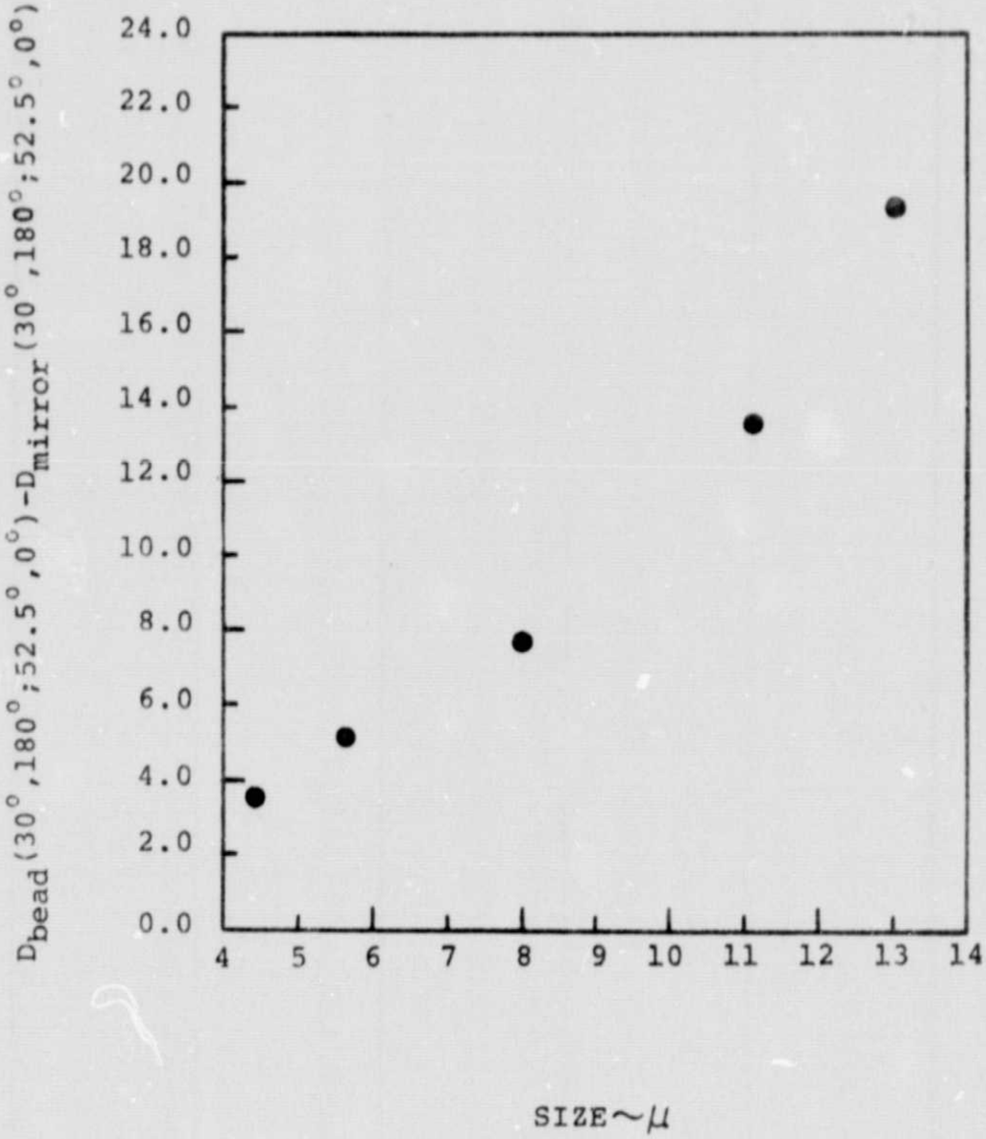

Figure 12 **Measurements of Scatter for θ=52.5° and $\emptyset$=0°.**

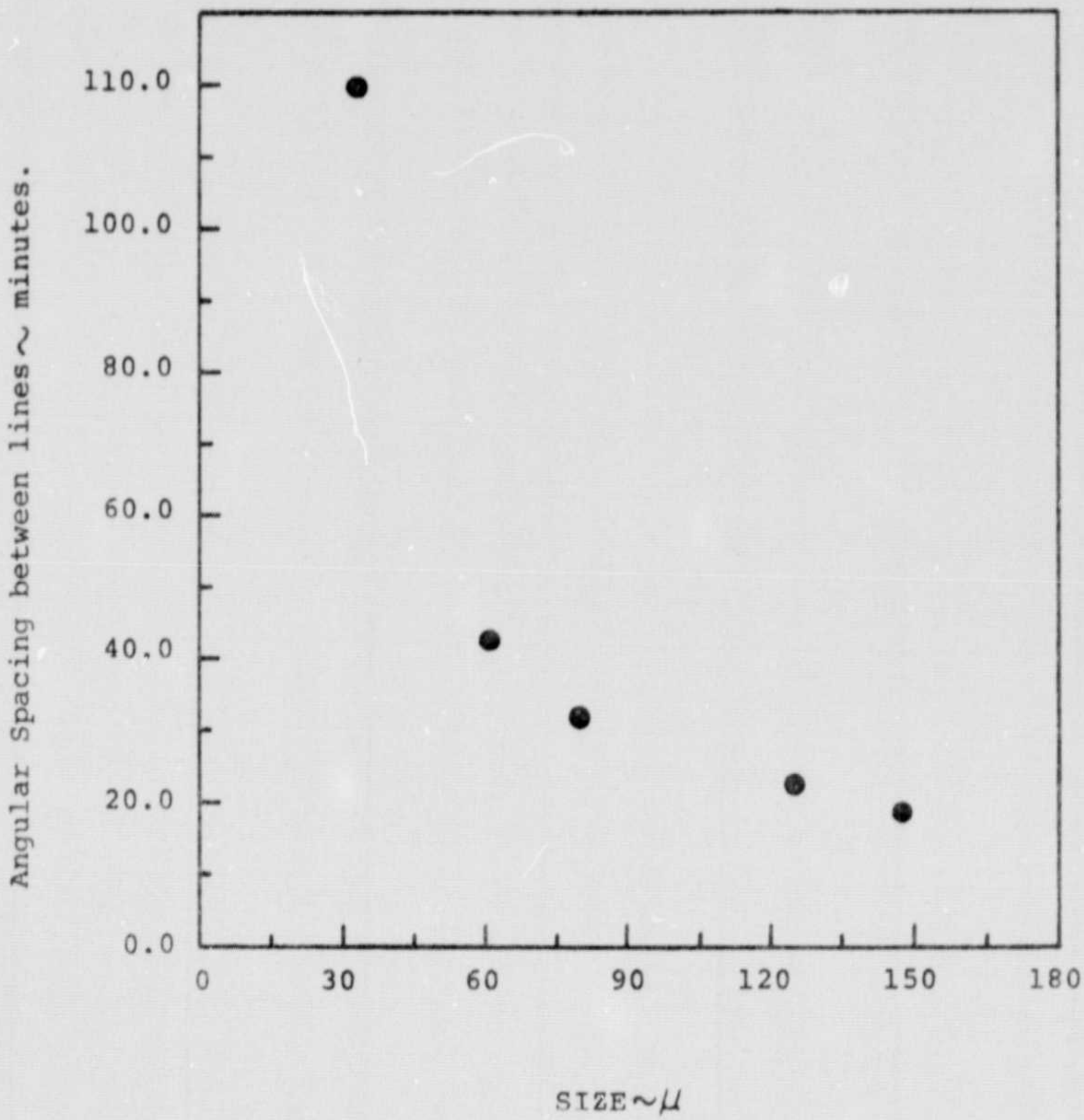

Figure 13 **Correlation of Angular Spacing between lines of Figure 3 and size of particles.**

TABLE 1

SCATTER OF MIRROR AT 0.6238 MICRONS*
D(30,180;θ,φ)

θ	φ=0	φ=180
0	0.625	0.625
5	0.895	-----
10	1.417	0.352
15	1.745	-----
20	3.780	0.304
36	-----	0.239
40	4.065	-----
45	3.058	0.194
50	3.160	-----
55	2.965	0.196
60	0.847	-----
65	-----	0.190
70	0.418	-----
75	-----	0.167
80	0.307	-----
85	0.290	0.150

* LASER

TABLE 2

SCATTER OF GLASS BEAD SIZE 135 MICRONS AT 0.6238 MICRONS*
D(30,180;θ,φ)

Θ	φ=0	φ=180
0	2.525	2.525
10	7.728	7.310
20	11.460	11.730
35	-----	199.930
40	95.850	13.790
45	-----	147.470
50	40.020	56.700
55	28.020	19.700
60	15.410	2.890
65	18.167	2.570
70	1.106	1.830
75	12.340	1.890
80	12.050	1.520
85	11.900	1.730

* Laser, solid angle of detector was 0.00076 sterians

TABLE 3

SCATTER OF GLASS BEAD SIZE 60 MICRONS AT 0.6238 MICRONS
$D(30,180;\theta,\phi)$

ϕ	ϕ=0	ϕ=180
0	1.640	1.640
10	4.163	4.400
20	8.920	8.330
25	37.400	-----
35	-----	6.130
40	24.280	14.790
41	19.440	-----
42.5	29.310	-----
43	19.360	-----
45	25.680	6.000
50	-----	8.740
50.5	7.790	-----
53	13.960	-----
55	11.440	3.230
60	4.310	3.330
65	5.647	2.740
70	6.700	2.090
75	6.720	1.530
80	10.860	1.500
85	5.990	1.500

* Laser, solid angle of detector was 0.00076 sterians

TABLE 4

SCATTER OF GLASS SPHERICAL BEADS WITH LASER
MEASURED WITH 1P28A P.M.T.*

θ	DIAM	DIAM
0.0	3.620	3.820
2.5	3.695	4.215
5.0	5.507	4.307
7.5	3.000	4.150
10.0	5.282	5.532
12.5	5.078	4.178
15.0	6.216	5.716
17.5	6.657	4.857
20.0	6.819	6.169

* $\psi=30^0$, $\phi=0^0$ $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$.

Note: Diameters are in microns.

TABLE 5

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 106	θ	DIAM 118
0.0	1.000	0.0	1.975
5.0	1.359	5.0	2.163
6.5	1.740	9.7	3.710
7.1	1.306	13.0	2.979
11.0	6.431	20.0	5.653
15.0	4.667		
20.0	5.313		

θ	DIAM 127	θ	DIAM 136
0.0	2.170	0.0	2.470
1.6	2.089	5.0	3.978
5.0	2.433	8.1	7.721
7.0	2.870	9.7	7.160
10.9	7.623	15.0	11.277
12.8	4.982	20.0	9.973
18.2	6.570		
20.0	6.183		

* $\psi=30^0$, $\phi=180^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 6

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 55	θ	DIAM 57
0.0	0.570	0.0	0.390
1.5	0.545	5.0	0.474
5.0	0.730	11.5	1.898
9.2	1.132	13.0	1.224
12.0	0.907	20.0	1.753
15.0	1.137		
20.0	1.363		

θ	DIAM 62	θ	DIAM 88
0.0	0.826	0.0	1.225
4.8	0.920	5.0	1.416
8.5	0.878	10.0	1.692
11.5	0.887	15.0	2.170
13.2	1.314	19.0	2.275
20.0	1.183	20.0	2.223

* $\psi=30^0$, $\phi=180^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 7

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 55
0.0	0.341
45.0	1.760
48.0	2.109
52.5	1.000
60.0	0.802
65.0	o.688
70.0	0.709
75.0	0.848

* $\psi=30^0$, $\phi=180^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 8

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 79	θ	DIAM 110
0.0	1.420	0.0	1.480
45.0	12.080	45.0	46.970
50.0	4.480	50.0	17.669
55.0	1.689	55.0	4.526
60.0	0.926	60.0	1.708
65.0	0.654	65.0	0.865
70.0	0.595	70.0	0.739
75.0	0.500	75.0	0.818

θ	DIAM 127	θ	DIAM 134
0.0	1.492	0.0	23.740
45.0	6.433	45.0	64.870
50.0	62.970	50.0	26.129
55.0	7.100	55.0	7.636
60.0	3.089	60.0	2.796
65.0	1.117	65.0	1.180
70.0	0.781	70.0	0.820
75.0	0.704	75.0	0.818

* $\psi=30^0$, $\phi=180^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 9

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.

θ	DIAM 87
0.0	1.000
20.0	4.030
45.0	21.470
50.0	9.030
55.0	3.336
60.0	1.430
65.0	0.840
70.0	0.550
75.0	0.500

* $\psi=30^0$, $\phi=180^0$, $\zeta= 180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 10

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 61	DIAM 81	DIAM 117
0.0	0.350	0.640	1.155
2.5	0.330	0.670	1.135
5.0	0.313	0.661	1.171
7.5	0.359	0.725	1.400
10.0	0.427	0.887	1.367
12.5	0.472	0.830	1.430
15.0	0.498	0.888	1.488
17.5	0.567	0.960	1.560
20.0	0.529	0.650	1.491

* $\psi=30^0$, $\phi=0^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 11

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 48	DIAM 134
0.0	0.313	1.440
2.5	0.314	-----
5.0	0.313	1.441
7.5	0.311	-----
10.0	0.322	1.922
12.5	0.319	1.970
15.0	0.372	1.932
17.5	0.300	2.340
20.0	0.175	2.841
37.5	3.980	32.460
40.0	2.060	29.550
42.5	3.460	29.810
45.0	4.320	28.010
47.5	4.240	24.400
50.0	3.680	22.760
52.5	3.600	19.500
55.0	2.850	18.870
60.0	2.331	16.920
62.5	1.995	14.100
65.0	2.099	13.200

* $\psi=30^0$, $\phi= 0^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.

TABLE 11 (cont)

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 48	DIAM 134
67.5	1.862	-----
70.0	1.690	-----
72.5	1.352	-----
75.0	1.176	-----

TABLE 12

SCATTER OF GLASS SPHERICAL BEADS WITH QUARTZ LAMP, MEASURED WITH 1P28A P.M.T.*

θ	DIAM 61	DIAM 85	DIAM 113
37.5	8.830	13.300	26.760
40.0	7.090	11.170	21.580
42.5	7.710	10.950	22.140
45.0	7.810	11.900	21.200
47.5	6.980	11.260	19.690
50.0	5.660	9.560	17.690
52.5	4.890	7.800	14.400
55.0	5.800	7.760	11.660
57.5	4.531	5.361	9.961
60.0	4.210	7.220	12.690
62.5	3.551	5.301	12.851
65.0	3.425	6.335	11.645
67.5	3.232	5.252	10.292
70.0	2.930	4.780	8.530
72.5	2.540	4.540	8.330
75.0	2.376	4.086	5.316

* $\psi=30^0$, $\phi=0^0$, $\zeta=180^0$

Relative to mirror reflectance at $\theta=0^0$

Note: Diameters are in microns.